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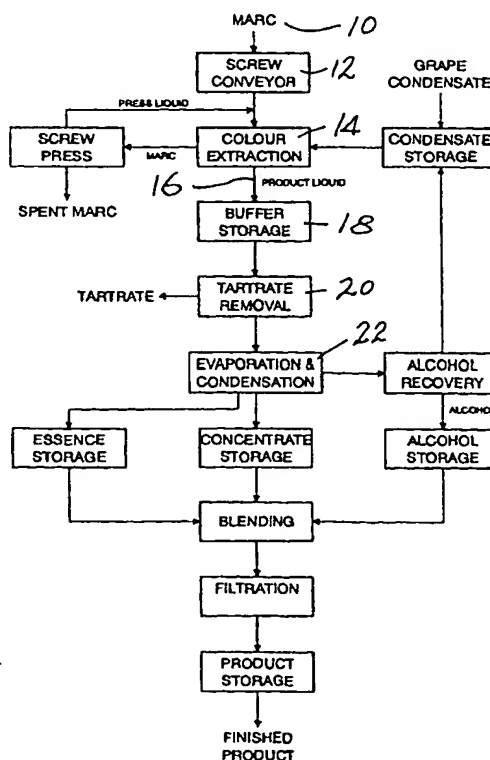
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(21) International Application Number: PCT/AU98/00594 (22) International Filing Date: 28 July 1998 (28.07.98) (30) Priority Data: PO 8301 29 July 1997 (29.07.97) AU (71) Applicant (for all designated States except US): TARAC DISTILLERS PTY. LTD. [AU/AU]; Barossa Valley Way, Nuriootpa, S.A. 5355 (AU). (72) Inventor; and (75) Inventor/Applicant (for US only): KLINGNER, Kenneth, Em [AU/AU]; 207 Murray Street, Tanunda, S.A. 5352 (AU). (74) Agent: COLLISON & CO.; 117 King William Street, Adelaide, S.A. 5000 (AU).		(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, HR, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG). Published <i>With international search report.</i>

(54) Title: PROCESS FOR THE RECOVERY AND IMPROVEMENT OF WINE EXTRACT AND DERIVATIVES FROM GRAPE MARC

(57) Abstract

A method of processing extracts from grape marc which includes the steps of obtaining from an extract of marc through the use of mixing and agitating marc with water and then including a subsequent step where the material is concentrated by evaporation under reduced pressure and increased temperature to result in separation of some water, alcohol and volatile aromas from the marc extract.



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PROCESS FOR THE RECOVERY AND IMPROVEMENT OF WINE EXTRACT AND DERIVATIVES FROM GRAPE MARC

FIELD OF THE INVENTION

This invention relates to a method of assisting in improvement of wine
5 characteristics and the recovery of materials from grape marc.

The process can be used in particular to improve red wine and to provide a concentrated anthocyanin product (containing no alcohol) from red grape marc.

The invention is not in its broadest form limited to red grapes or grapes.

10 BACKGROUND ART

To produce wine, juice is extracted from grapes by pressing and is collected for further processing after it has been separated from the marc which is to say the skins and stems, in the case of white wine and subsequent to being left on the marc or being mixed with the marc in the case of red wine.

15 Red grape marc however have recoverable components. In red grape marc, it is conventional to ferment to some selected degree the juice with the marc and thus that marc also contains a significant proportion of alcohol. This would normally be lost upon disposal of the marc. This marc has hitherto been distilled to extract alcohol, with some 50 to 60 litres of alcohol
20 extracted from 1000 kg of marc. Whilst distilling recovers the alcohol it does not recover any of the other wine extracts and/or derivatives such as colour or tannins.

It is known to recover wine extract and/or derivatives from marc by liquid infusion of the marc and then using reverse osmosis and ultrafiltration
25 membranes to separate useful components. This selectively separates to some extent some components such as alcohol and essence.

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Membrane usage is however relatively costly and replacement costs are relatively high. Further, membranes are prone to blockage for instance due to tartrate crystal build up created during the process. Furthermore, membranes used in a reverse osmosis arrangement can separate some of the alcohol and water from an extract but the process is inherently unable to achieve higher levels of concentration required to develop colour and other characteristics of the extract for most effective blending or other usage.

The ability to extract as much colour and flavours as possible is of very high economic significance since the recovered wine extract may then be used as an additive to improve existing wine or to increase yield from an original quantity of grapes.

Further, if an extract can be certified to be of only grape origin then the wine extract can be mixed with existing wine to enhance their colours and/or flavours, whilst still satisfying regional regulatory requirements which are in force in some countries of the world including Australia.

It is an object of this invention to enable a more useful material to be obtainable from grape marc than has hitherto been available and at the least provide a useful alternative to current processes which can be technically and economically of benefit.

DISCLOSURE OF THE INVENTION

According to this invention there is proposed a method of processing extracts from marc which includes the steps of obtaining an extract of marc through mixing and agitating with water and then including a subsequent step where the material is concentrated by evaporation.

In preference the evaporation is effected in a way that will result in separation of some water, alcohol and volatile aromas from the marc extract.

In preference there is proposed a method to extract with water infusion from marc the method including the steps of:- mixing and agitating marc with water and collecting a resulting aqueous solution and mixture as an extract; and concentrating said resultant extract by thermal evaporation under

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reduced pressure the temperature and pressures being selected to separate aromas, some water and some alcohol from colouring material.

In preference the method further includes the steps of:-

- 5 after mixing and agitating marc with water and collecting a resulting aqueous solution and mixture as an extract;
then separating coarse solids from the extract;
chilling the resultant material to effect a crystallization of tartrate;
and removing any formed tartrate crystals; prior to
10 concentrating said resultant extract by thermal evaporation.

In preference the method further includes the steps of:-

after concentrating said resultant extract, recovering alcohol and water by partial condensation of the vapours.

15

In preference the method further includes the steps of:-

recovering of alcohol and essence components remaining in condensates by thermal evaporation at low pressure otherwise referred to as flash stripping; and

- 20 chilling of the extract and alcohol and/or essence streams.

In a further form of this invention this could be said to reside in preference in a method to recover from marc useful materials including the steps of:-

- 25 mixing and agitating grape marc with heated water and collecting the resulting aqueous solution and mixture as an extract;
cleaning of larger solids from the extract;
chilling said extract whereby to effect tartrate stabilising of said extract, and then removing tartrate crystals from the extract by centrifuge;
concentrating the resultant extract material by thermal evaporation under
30 reduced pressure by separating thereby as a vapour some water, alcohol and aromas from anthocyanins;
recovering alcohol and water from the said vapour by partial condensation of the vapour;
separating alcohol from the water in the condensate; and
35 chilling of the resultant anthocyanins extract and alcohol and essence streams and blending back together of the concentrated anthocyanins with alcohol and aromas.

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Thus, in preference the method includes extraction, stabilisation, solids removal, concentration and filtration. Extraction using hot water at approximately 50 degrees Celsius diffuses alcohol and grape anthocyanins (red colour) into grape water with resultant extracted liquid containing
5 alcohol, grape anthocyanin and other dissolved and suspended solids. The remaining marc residue such as skins and stalks is then disposed of by standard means.

The extracted liquid is then chilled to bring the temperature down from the 50 degrees Celsius in order to precipitate potassium hydrogen tartrate, the level
10 of chilling and time determined by the level of tartrate in the extract. Centrifusion is then employed to separate precipitated tartrates and other suspended solids from the extracted liquid which is then pumped into a falling film long plate evaporator with an aroma recovery system designed to operate at temperatures of some 40 degrees Celsius to avoid degradation of
15 the product.

The grape marc is first mixed with added warm water in a known method to produce a liquid extract that will contain any alcohol and colouring material from the grape marc. The added water is chosen in preference to be grape water to ensure that all of the products produced from this process satisfy
20 any label integrity requirements concerning allowable additions which can inhibit the addition of water to wine. Any residue that then leaves the extractor can be subjected to a squeezing so as to be in as dry condition as possible and may then go for further processing to recover any residual alcohol.

25 In preference the evaporative system is adapted to use reverse-feed multi-effect evaporators.

In preference the process is a closed-loop process operating under vacuum provided by vacuum pumps.

In preference the condensed alcohol/essence liquid is used as a sealing
30 fluid in any vacuum pump.

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Preferably one uses a flash evaporator which is to say a low pressure with moderate temperatures to recover components of alcohol and flavour components after the evaporation step.

BRIEF DESCRIPTION OF THE DRAWINGS

- 5 The present invention will now be described, by way of example only, with reference to the accompanying drawings in which:-

FIG 1 is a flow chart of the process in accordance with a first aspect of the present invention;

- 10 FIG 2 is a flow chart of the production of red wine from grape marc to produce a colour density of 15-30 absorbance units and alcohol concentration of some 8-12% V/V;

FIG 3 is a flow chart of the production of red wine from grape marc to produce a colour density above 30 absorbance units and alcohol concentration of up to 14.5% V/V; and

- 15 FIG 4 is a flow chart of the production of red wine from grape marc to produce a colour density above 30 absorbance units and no alcohol.

BEST MODE FOR CARRYING OUT THE INVENTION

- Referring to the drawings, grape marc 10 is first fed via a suitable means such as a screw conveyor 12 to an extractor 14 to produce with the addition
20 of heated water derived from a grape source at a temperature of approximately 50 degrees Celsius an extract 16. When the extract 16 leaves the extractor 14, the water contains both dissolved materials and suspended fine and coarse solid matter which is removed prior to further treatment. In this embodiment, the process used is a specific hydrocyclone which
25 removes some of the particles by centrifugal action. The extract is then further refined using a brush strainer to remove remaining particles.

The extract is then chilled in a chiller unit to reduce the temperature of the marc to around 10 degrees Celsius and it is then stored in buffer storage 18.

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The chilled extract then undergoes tartrate removal 20 where the extract is transferred to a tartrate stabilisation tank in which tartrates present from the grape in the original extraction are allowed to come out of solution at the cooler temperature and this is normally in the form of crystals. In addition, these tartrates are a valuable side product. Additionally, by removing them before the evaporation process, concentration and quality of the extract is improved. The tartrates settle out over time in the stabilisation tank and are progressively removed and collected by using a hydrocyclone treatment followed by centrifuge which centrifugally spins out the crystals from the extract. The tartrate crystals are then treated separately, whilst the extract is transferred to an evaporator feed tank.

The extract is then processed both to concentrate it so as to reduce the quantity of any water which of course was added during the extraction stage which produces a required colour intensity in a finished product.

15 The process step of concentration of the extract is the use of a thermal process of evaporation 22, using film type evaporators, with steam as the service media and the evaporation is effected under reduced pressure which allows for greater evaporation at lower temperatures.

20 The evaporator type used in the embodiment is a single pass long plate system which has relatively short residence time and which produces an extract with the desired characteristics of colour and flavour.

The evaporators are in one case a single and in an alternative embodiment a multiple effect, with an operating mode determined by factors including the characteristics desired in the extract and any operating economy which improves multiple effect operation.

In one mode, the evaporator is a single pass operation with no recirculation of extract so as to achieve the shortest residence time and least thermal degradation of the extract.

30 Alternatively one may apply limited recirculation so as to increase the wetting rates of the heating surfaces and also to produce different characteristics in the finished extract if required.

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For example, where one wishes to obtain a high concentration (say 8:1 or better) this will usually require some recirculation to retain wetting rates for the extract.

- 5 A further embodiment is the use of reverse feed in the multi-effect evaporators wherein the extract is introduced initially to the final effect claudria in order to recover the maximum yield of alcohol/essence from the first vapours produced off the extract.

In an alternative mode, there is a forward feed system in which the volatiles are recovered from the vapours of the first or second effect.

- 10 (The evaporative process maximises yield of volatile compounds of alcohol and essence/flavour bearing components and recovers them in concentrated form. Concentrations of alcohol greater than 60% may be achievable. The system may also be adjusted to lower levels as required for add-back to the concentrated product to produce wine with alcohol levels as
15 required by the processor. Concentration of extract of some 6:1 are achievable by the evaporation process.)

- The next stage is the recovery of the alcohol/essence from vapours. First vapours evaporated off the extract contain the bulk of the alcohol and flavour volatiles. These are fed into a partial condenser in which the condensing
20 process may be adjusted to condense whatever portion of the water phase in the vapour is required to be condensed. For example 90% of the water vapour is condensed at the saturation temperatures of the water vapour as given in known steam tables, the remaining 10% of uncondensed steam vapour will retain almost all of the volatile compounds, which require a lower
25 condensing temperature according to the properties of the alcohol/aroma compounds. This then results in a much stronger volatile bearing vapour stream leaving the primary condenser. This remaining vapour stream is then fully condensed using cooling water and refrigerant to give a strong alcohol/essence stream.

- 30 The vapour in the primary condenser is maintained under vacuum using a liquid ring or other vacuum pump and the condensing temperature may be adjusted as required by control of a vacuum pump performance.

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In this embodiment, the condensed alcohol/essence liquid is used as the seal water in the vacuum pump which is ideally of a sanitary construction. The discharge from the vacuum pump, which is liquid and air remaining from the primary condenser, is discharged into a separation vessel and vented to
5 a packed scrubbing column. The packing is washed with a flow of chilled liquid, which can be condensate off the evaporator or grape water from another source. This then recovers any remaining components of alcohol/essence from the air before discharge into the atmosphere. The stream of low recovery liquid can be returned to the process in various ways
10 such as for example by flashing to re-introduce the volatiles to the system or alternatively by returning the liquid to the base of the primary condenser for recovery in the next part of the process.

The next stage of the process is the recovery of alcohol/essence from condensates. In the base of the primary condenser there are condensates
15 from the evaporation of the wine extract, which contain substantially all of the remaining volatile components which have not been collected, in the concentration system previously described, apart from small losses in the air discharged from the scrubbing column. The remaining components of alcohol and flavour compounds are recovered in a flash evaporator. Other
20 types of evaporators could be used for this recovery process, however, the flash evaporator is one of the more economic and effective methods.

The flash evaporator may be use either a single effect operating mode or multi-effect determined by the capacity of the plant and operating cost comparisons. Some other embodiment can employ double or triple effect
25 systems, although more or less effects may be used with acceptable results.

For the evaporator, a plate and a frame design is used for optimal results although a tubular design may equally well be used. When using a double effect plant, the condensate is pumped to the first effect and circulated through the first effect plate heat exchanger where it is heated by steam or
30 other medium to a temperature, usually in the range of 60 to 95 degrees Celsius. The condensate then enters a vacuum separator, maintained by a vacuum pump at a pressure below the saturation temperature of the condensate, via a throttling valve. The reduction in pressure causes vapour

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to be liberated from the condensate by flash evaporation and this vapour is taken to the heating side of the second effect.

5 The condensate remaining in the first effects evaporator is pumped to the product side of the second effect where it is heated by the vapour from the first effect to a temperature approaching the vapour temperature. The condensate is then admitted via a throttling valve to the second effect separator which is maintained at a pressure below the saturation temperature of the condensate by the same vacuum pump.

10 The condensed vapours from each effect are combined and this now represents concentrated volatile components. In order to obtain a sufficiently high level of evaporation to recover the volatiles recirculation of condensate is applied around each effect which increase the amount of evaporation.

15 A feature is that the operating mode of the flash evaporator may be varied to provide the best results both in terms of quality and yield. For example, the vapour from the first effect can be used as feed to the second effect to increase the concentration. In a further embodiment of a triple effect plant, the vapours condensed off the first two effect can be combined to form the feed to the third effect, or any combination of feed systems can be used to maximise quality and yield. The flash system herein described can recover
20 alcohol and other components, which may be 4 to 5% v/v in the initial condensate, and concentrate them to levels as high as 30-40% v/v for example for add-back into the total volatiles recovery system.

25 The extract concentrate off the evaporator is then chilled and taken to a concentrate tank, usually with a chilled jacket. The combined streams of condensed and chilled volatile components are taken to a volatiles recovery tank, usually fitted with a chilling jacket. The contents of the two tanks may then be blended to produce a wine product having the desired alcohol content. This can be achieved, for example, by the use of metering pumps.

30 It is also to be understood that this process can be used effectively on white wine marc, but as no fermentation is involved, the value of the final product is reduced by the absence of alcohol.

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This process does not constitute the fortification of wine since there is no spirit added from external sources. It is also not a distillation process, even though in some applications there may be surplus alcohol from the process.

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CLAIMS

1. A method of processing extracts from marc which includes the steps of obtaining an extract of marc through the use of mixing and agitating with water and then including a subsequent step where the material is
5 concentrated by evaporation.
2. A method as in claim 1 further characterised in that the evaporation is effected in a way that will result in separation of some water, alcohol and volatile aromas from the marc extract.
3. A method of processing extracts from marc the method including the
10 steps of:-
mixing and agitating marc with water and collecting a resulting aqueous solution and mixture as an extract; and concentrating said resultant extract by thermal evaporation under reduced pressure the temperature and pressures being selected to separate aromas, some water and some alcohol
15 from colouring material.
4. A method of processing extracts from marc as in the immediately preceding claim further characterised in that the method further includes the steps of:-
20 after mixing and agitating marc with water and collecting a resulting aqueous solution and mixture as an extract;
then separating coarse solids from the extract;
chilling the resultant material to effect a crystallization of tartrate;
and removing any formed tartrate crystals; prior to
25 concentrating said resultant extract by thermal evaporation.
5. A method of processing extracts from marc as in either one of two the immediately preceding claims further characterised in that the method further includes the steps of:-
30 after concentrating said resultant extract, recovering alcohol and water by partial condensation of the vapours.

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6. A method of processing extracts from marc as in one of the preceding claims 3, 4, or 5 further characterised in that the method further includes the steps of recovering of alcohol and essence components remaining in condensates by thermal evaporation at low pressure and chilling of the extract and alcohol and/or essence streams.

7. A method to recover from marc useful materials including the steps of:-mixing and agitating grape marc with heated water and collecting the resulting aqueous solution and mixture as an extract;
10 cleaning of larger solids from the extract;
chilling said extract whereby to effect tartrate stabilising of said extract, and then removing tartrate crystals from the extract by centrifuge;
concentrating the resultant extract material by thermal evaporation under reduced pressure by separating thereby as a vapour some water, alcohol
15 and aromas from anthocyanins;
recovering alcohol and water from the said vapour by partial condensation of the vapour;
separating alcohol from the water in the condensate; and
chilling of the resultant anthocyanins extract and alcohol and essence
20 streams and blending back together of the concentrated anthocyanins with alcohol and aromas.

8. A method as in any one of the preceding claims further characterised in that evaporation in at least one of the steps is by way of use of a reverse-feed multi-effect evaporators.

25 9. A method as in any one of the preceding claims further characterised in that the method is operated as a closed-loop process operating under a low pressure provided by vacuum pumps.

10. A method as in preceding claim 9 further characterised in that condensed alcohol/essence liquid is used as a sealing fluid in any vacuum
30 pump.

11. An extract resulting from a method as characterised in any one of the preceding claims.

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12. Wine to which an extract as characterised in claim 11 has been added.

5 13. A method of extracting and treating marc substantially as described in the specification with reference to and as illustrated by the accompanying drawings.

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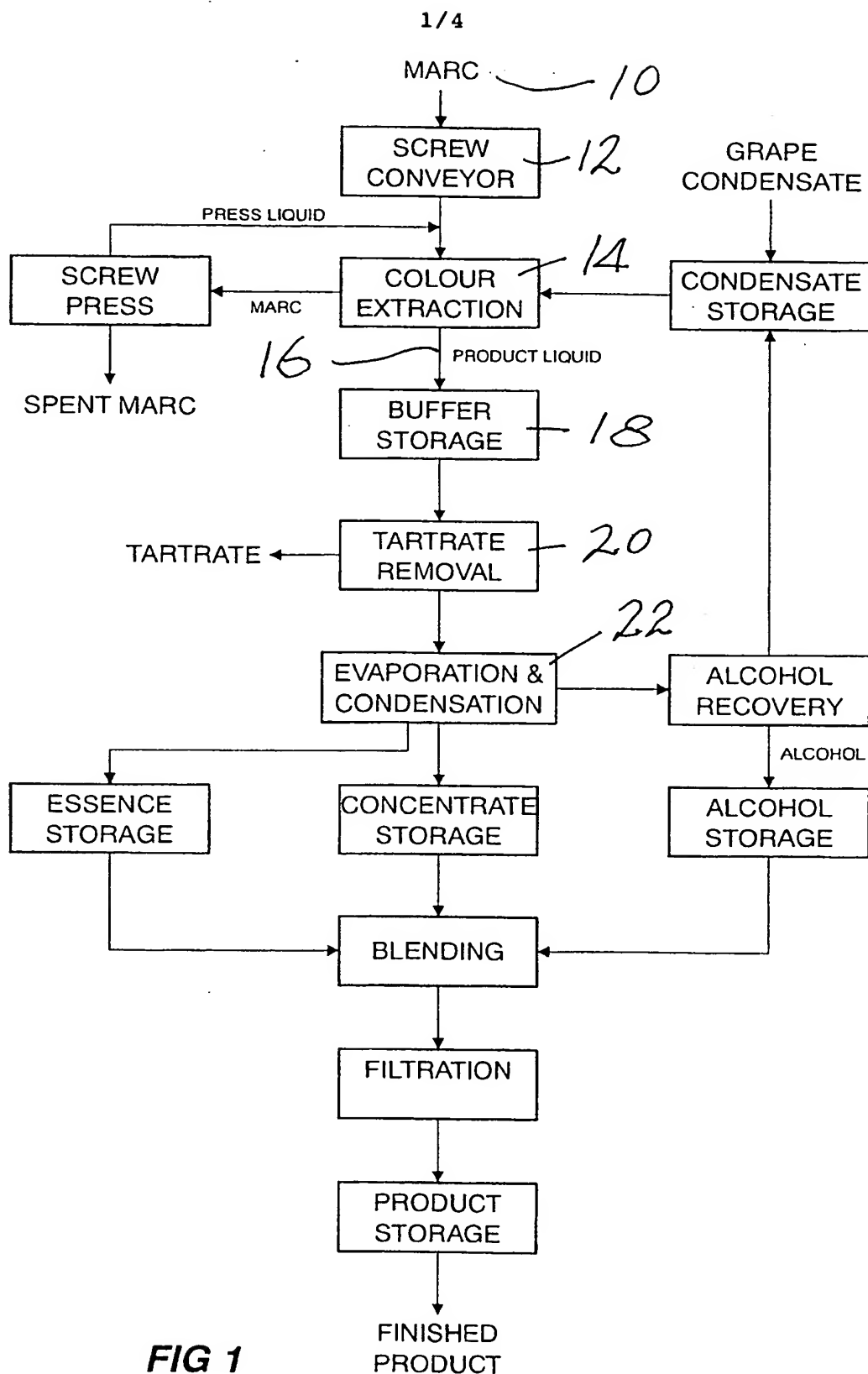
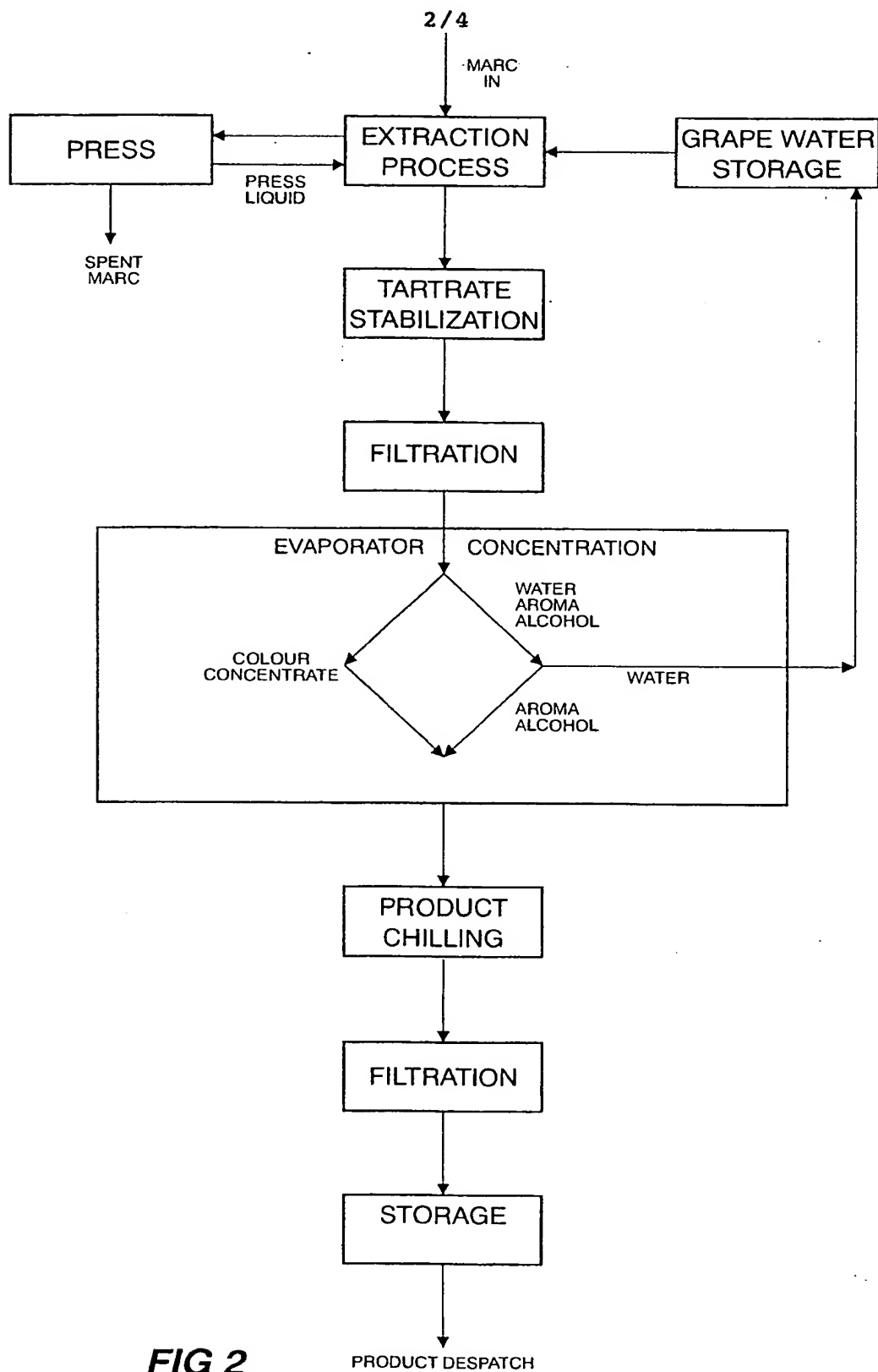


FIG 1

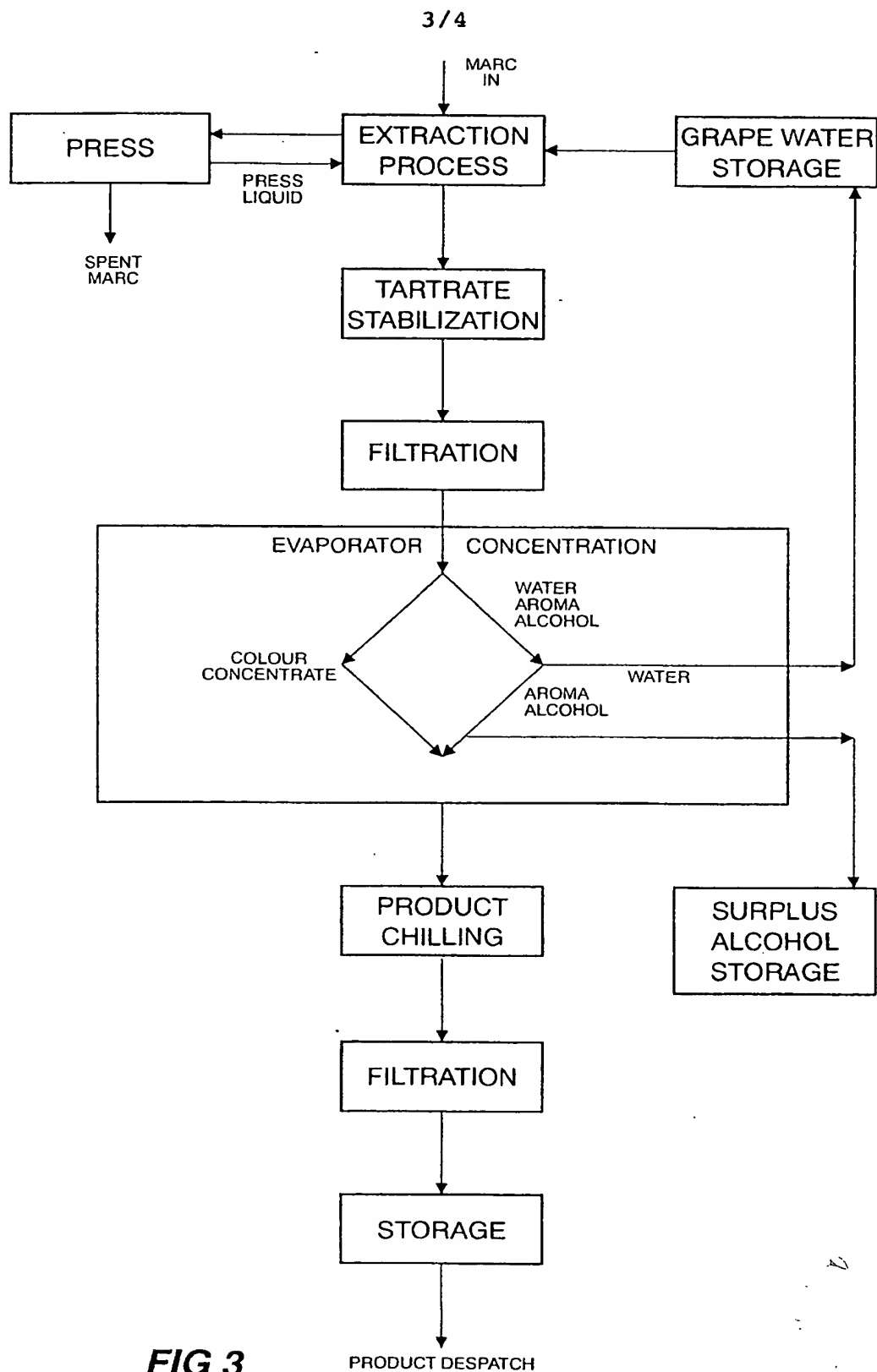
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**FIG 2**

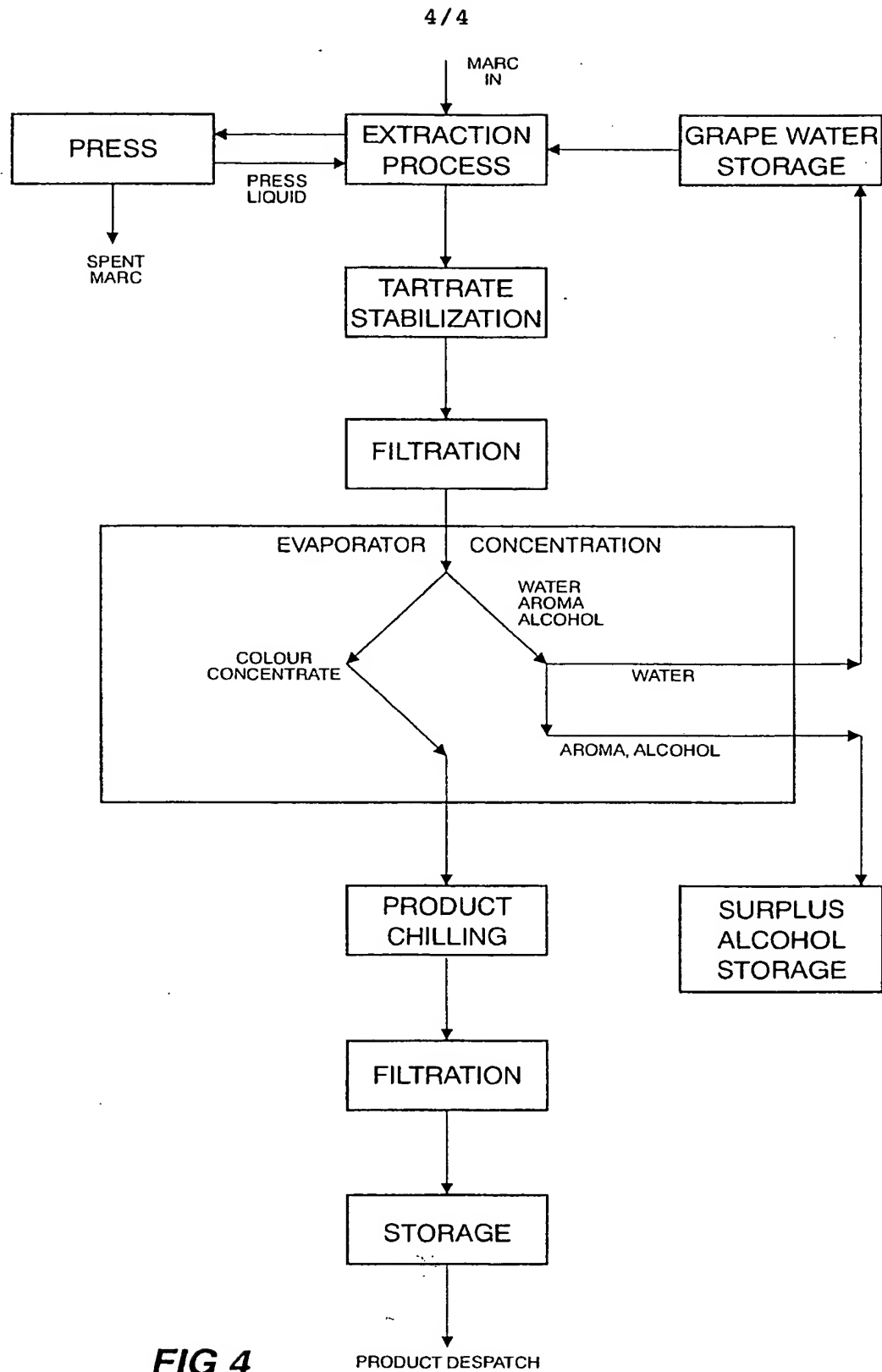
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**FIG 4**

INTERNATIONAL SEARCH REPORT

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A. CLASSIFICATION OF SUBJECT MATTER		
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X	FR A 2378070 (SOCIETE COOPERATIVE ROUSSILLON ALIMENTAIRE - LA CATALANE) 18 August 1978 See whole document especially example 4	1-11,13
X	DE,A, 3038198 (ROUSSEL-UCLAF) 7 May 1981 See whole document especially page4, 9-16 and example 1)	1-11,13
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Date of the actual completion of the international search 8 October 1998		Date of mailing of the international search report 13 OCT 1998
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C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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A	US A 4500556 (LANGSTON M.S.K.) 19 February 1985	
A	Chemical Abstracts volume 93 abstract no 130587 JAPAN KOKAI TOKKYO KOHO JP A 55081580 (KIKKOMAN SHOYU CO Ltd. Japan) 19 June 1990	

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.
PCT/AU 98/00594

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Patent Document Cited in Search Report				Patent Family Member			
FR	2378070	ES	466189	PT	67493		
DE	3038198	BE	885574	CH	651209	FR	2466989
		GR	70759	IT	1144015	PT	71883
US	4500556	DE	3310340	GB	2118566		
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